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भारतीय मानक

पर्यावरण नमूनों में रेडियोन्युक्लाइड — आंकलन की पद्धतियाँ

भाग 5 नमूना चयन की पद्धतियाँ

Indian Standard

RADIONUCLIDES IN ENVIRONMENTAL SAMPLES — METHODS OF ESTIMATION

PART 5 SAMPLING

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Nuclear Materials for Peaceful Applications Sectional Committee, CHD 30

FOREWORD

This Indian Standard (Part 5) was adopted by the Bureau of Indian Standards, after the draft finalized by the Nuclear Materials for Peaceful Applications Sectional Committee had been approved by the Chemical Division Council.

Collection of samples for radioactivity surveillance of environment depends on the purpose for which the results shall be used. Selection of sample type, sampling location, frequency of sampling and sample size to be used for analysis are all depend on the objectives of monitoring work. The results obtained may be used for checking regulatory compliance or for assessing the spatial and temporal changes in radioactivity levels or for environmental research such as radionuclide migration modelling, dating, etc. For obtaining dependable results, special attention is required in sampling, sample processing and preservation and also to prevent any contamination.

ISO has published ISO 18589-2 : 2007 'Measurement of radioactivity in the environment — Soil — Part 2: Guidance for the selection of the sampling strategy, sampling and pretreatment of samples'. However, there is no ISO Standard encompassing all other environmental matrix. The document is prepared based on the indigenous data available and taking assistance from the following documents:

a) BARC report BARC/1992/PD/OO2, Analytical procedures, Manual of Health Physics Division, BARC, 1992.
b) IAEA Technical Report Series No. 295, Measurement of Radionuclide in food and the environment (A guide book), IAEA, 1989.
c) EML Procedures Manual Report HASL -300; 26th Edition; Environmental Measurements Lab., N.Y. USA, 1983.

The other parts in this series are:

Part 1	Gross beta activity
Part 2	Gross alpha activity
Part 3	Uranium
Part 4	Radium

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

RADIONUCLIDES IN ENVIRONMENTAL SAMPLES — METHODS OF ESTIMATION

PART 5 SAMPLING

1 SCOPE

This standard (Part 5) prescribes the methods of sampling for determination of radionuclides in environmental samples.

2 GENERAL ASPECTS OF SAMPLING AND SAMPLE PRESERVATION

2.1 Sample Collection

The sample collected should be representative with respect to area, time and the matrix being sampled. Radioactive element is often present in sub microgram amounts and a significant fraction may be lost by adsorption on the surface of containers used in the sampling. The sample should be collected in adequately cleaned plastic bottles for liquid samples, bags and wide mouth bottles for solid samples. A record of sample identification number, date of sampling, sample description, location and fresh weight should be noted on the sample container and also in a register maintained at the Laboratory. The minimally required sample amount depends primarily on the concentration of the analytes present in sample matrices. The sample volume should be sufficient to perform all required laboratory analyses with an additional amount remaining to provide for analysis of QA/QC including duplicates and spikes. For heterogeneous samples, a larger portion is generally required to be a representative of the actual sample variations such that possible biased results can be minimized. This larger portion of the collected sample is then homogenized thoroughly followed by subsampling. Taking too large or too many samples can be avoided because of its storage (cold room and freezer), transportation, and disposal costs.

2.2 Sample Storage

The collected samples shall be properly stored to avoid degradation, spoiling or decomposition and also to avoid contamination, until it is taken up for analysis. In case of analysis of volatile, or short lived (like I-131 evaluations) radionuclides, samples should be analysed at the earliest possible. Sample storage for short duration may require refrigeration, freezing, or the addition of a preservative such as sodium bisulphite, alcohol or formalin for preservation of biological samples.

In case of the liquid samples, the loss of radionuclides due to absorption on the walls of the container can be minimized by the addition of concentrated hydrochloric acid (HCl) for radio cesium and mixture of conc. nitric acid (HNO_3) or hydrochloric acid (HCl) to obtain pH < 2 for all other radionuclides. Stable element carriers of the radionuclide of interest or some complexing agent may also be added. Sample storage area should be clean.

2.3 Pre-treatment of Samples

2.3.1 *Drying, Evaporation and Ashing*

Drying reduces the weight of the samples and also permits storage for a longer time. Drying may be done in an oven at a temperature ranging between 100 and 110°C for a sufficient period of time to give a constant dry weight of the sample. To prevent any loss of volatile radionuclides such as I-131, freeze drying can be adopted for analysis of radioactive constituents in easily volatilizing organic compounds. Freeze drying is time consuming and also expensive and as such it may be used only when it is essential.

The sample should be dried in an oven at 105 ± 5°C. Ashing the sample reduces the bulk of the sample considerably and helps in storing it for a long period of time. The temperature recommended for dry, ashing in muffle furnace is 450 ± 10°C. If the temperature of ashing goes beyond 500°C, there can be some loss of radiocesium and radioruthenium. The radioactivity contents in a sample should be expressed on dry weight basis rather than ash weight basis. Measurement of ash weight is necessary for calculations of radionuclide concentration. Platinum dish or platinum crucible are the best containers for ashing. Low carbon nickel dishes, crucibles or trays for radionuclide other than nickel (Ni) isotopes or silica (vitrified) dishes may also be used for ashing the environmental samples.

2.4 Homogenization

The homogeneity of the sample always plays a role when the material under investigation is not mixed thoroughly. Dried or ashed samples require homogenization before taking any portion of it for analysis. Homogenization can be carried out using mixer-blenders, ball mill or manually using mortar and pestle. Large quantities of powdered sample can be homogenized in a rotating stainless steel drum homogenizer.

3 SAMPLE AMOUNT

Amount of the sample to be taken for analysis depends on following two factors:

a) Fraction of the derived limit of radionuclide concentration that has to be measured.

b) The detection limit of the measuring instrument with the required confidence level.

Lower limit of detection (LLD) or minimum detectable activity (MDA) is defined as the smallest quantity of sample radioactivity that will yield a net count on which there is a predetermined level of confidence that radioactivity is present. For sample and background counting rates that are similar (as expected at or near the LLD) the smallest amount of radioactivity that has a 95 percent probability of being detected is,

$$\text{MDA (Bq/kg)} = \frac{2.71 + 4.66\sqrt{B}}{T \times E \times \gamma_{\text{abn}} \times (M \text{ or } V)}$$

where

B = counts;
T = counting time, in s;
E = counting efficiency;
$_{\text{abn}}$ = gamma abundance;
M = sample weight, in kg; and
V = sample volume, in l.

Example:

Say one wants to measure Cs-137 in food samples. Determine the optimum amount of sample, if 0.1 percent of derived maximum permissible concentration (MPC) of Cs-137 in food has to be measured. Derived MPC for Cs-137 in food is given as 1 000 Bq/kg. That is, if 1 Bq/kg is to be measured and the sample amount to be taken for this can be obtained by using the above relation and the following values for the factors:

0.1 percent of derived MPC = 1.0 Bq/kg, T = 3 600 S, B = 5 000 counts

E = 0.1 (10 percent), $_{\text{abn}}$ = 0.851 (85.1 percent)

$$\text{Then } M \text{ (mass of sample required} = \frac{2.71 + 4.66 \times \sqrt{5\ 000} \times 1\ 000}{3\ 600 \times 0.1 \times 0.851 \times 1.0}$$

that is, as per the above relation, 1 084 g of sample should be sufficient for analysis.

The sample amount should be selected on the basis of nuclide of interest having the lowest derived limit when analysis is required for various radionuclides.

4 SAMPLING AND SAMPLE PREPARATION (PROCESSING)

4.1 Type of Samples

The samples for radioactivity measurements can be any of the following environmental matrices:

a) Atmospheric;

b) Atmospheric fallout deposition or wet precipitation;

c) Terrestrial; or

d) Aquatic (fresh water or marine).

4.2 Atmospheric Sampling

4.2.1 Samples from the atmospheric air are classified into two categories, namely, gaseous and particulates. The equipments required for air sampling are:

a) Air suction pump;

b) Flow rate meter;

c) A time totalizing device; and

d) Filter (holder) head for particulates or adsorbent cartridge for gases.

4.2.2 Air should be pumped through the filter at known flow rate for a known period of time so that total volume of air sampled can be known and radioactivity concentration is reported per unit volume (Bq/m^3 or Bq/l).

4.2.3 In case of gaseous I-131 collection, either activated charcoal cartridge or charcoal impregnated filters are used as adsorbers. For the purpose of particulate collection, there are many varieties of filter media such as glass fibre filter paper, cellulose fibre filter papers, polycarbonate filters, etc. Pumps with lower flow rates (50 to 100 l/min) can be used for routine air sampling to obtain weekly averages. There are air sampling equipments available with constant flow rate or a flow integrating device to take care of the varying dust loads. Battery operated samplers can be used where electric mains supply is not available.

For sampling air particulates in emergency conditions, high volume air samplers (300 l/min) can be used. Though the sampling time is dependent on the expected activity level in any case, for rapid assay, it need not be more than 30 min.

4.3 Atmospheric Fallout Deposition or Wet Precipitation

4.3.1 Dry fallout deposition sample may be collected on a stainless steel or a plastic tray of known area. Normally this sample is collected on a monthly cumulative basis. Collection tray area of 1 m^2 is adequate. The fallout collectors should be kept in a completely open area without cover of trees or bushes.

4.3.2 For the collection of rain water, a wide diameter (15 cm) plastic funnel attached to a Plastic carboy (20 l) may be used. While sampling rain water for longer duration (weekly or monthly), stainless steel or plastic trays of 0.1 m^2 area with arrangement to drain the collected water to a container carboy may be used. Mixed bed ion exchange columns with known water collection area (area of the funnel kept on the top of the column) may also be used for weekly or monthly rain water sample collection. From the rainfall data and area of the collection, one can get the volume of rain water passed through the column. The rain water collectors also should be kept in an open area.

4.4 Samples from Terrestrial Environment

4.4.1 Soil, grass vegetation/vegetables, food crops, milk and meat are the important matrices from the terrestrial environment for the purpose of radiation exposure evaluation. Certain samples are good indicators of terrestrial radio activity deposition of particular radionuclides. For example, animal thyroids especially of goat/sheep are good concentrators of deposited I-131. Dung of the grazing cattle is a good indicator of pasture deposited radionuclides like plutonium. Leafy vegetation is good indicator of terrestrial deposited radionuclides.

4.4.2 Soil and soil radioactivity is measured to evaluate,

a) radionuclide deposited on land (which will give rise to direct radiation exposure);
b) availability of radionuclides for pick up by vegetables and crops grown over it; and
c) to estimate the leachable fraction of radionuclides to ground water.

For evaluating land deposition, soil has to be collected from undisturbed area on level ground and open terrain. An area of 1 m^2 has to be marked and all the soil up to a depth of 15 cm should be collected by digging with a small pick-axe. If there is grass growth over the land, the top grass has to be cut and removed before soil sampling. Root and mat portion can remain. For the purpose of evaluating radionuclide pickup by food crops, soil up to a depth of 5 to 10 cm has to be collected to cover the complete root zone.

Auger samplers can be used to collect soil core up to the required depth. Instead of digging with pick-axe, soil coring in about 10 places in 1 m^2 area up to the required depth can be carried out. The cores have to be mixed to make a single representative soil sample. Soil samples can be put in polythene bags and labeled properly. In the laboratory, samples may be dried, powdered and rocks and stone particles may be removed. Dry powder may be sieved through a 70 mesh sieve and this processed soil may be stored in wide mouthed polythene or plastic bottles.

4.4.3 *Grass*

Grass and any other type of animal forage are sampled from known land area. A 1 m × 1 m (1 m^2) area frame may be made from wooden strip and can be used to mark 1m^2 area on grass land. The frame may be kept on the grass land and all grass within the frame may be cut just 4 cm above ground so as to prevent any soil contamination on grass and also represent the portion normally used as fodder. If grass is grown only to a small height it can be cut just above ground avoiding soil contamination. In case of huge wide grass lands, samples from three to four locations from the same area may be composited to get a representative sample for the area. Grass can be collected in a polythene bag on which the sample identification number and other details shall be written. If the grass is green and not dry, the sample weight should be taken at the earliest.

For storage of green grass, perforated plastic bags may be used. If I-131 is to be estimated, the grass sample in the bag can be directly counted provided the instrument calibration exists. Otherwise grass should be chopped into small pieces, dried at 100 to 105°C, powdered in a mixer-grinder, and this dry powder used for radionuclide determination by gamma spectrometry. The dried grass can be dry ashed in a muffle furnace below 450°C or wet ashed using concentrated nitric acid (HNO_3) and hydrogen peroxide (H_2O_2) for further radiochemical analysis.

Sample weight, dry weight, and ash weights should be properly registered such that the result expressed as Bq/g ash can be converted for any required state of sample.

4.4.4 *Vegetables/Vegetation*

While sampling vegetables/vegetation from the field, a composite sample should be made by taking small and equal quantity of vegetables/vegetation from different places in the same area. A sample weight of about 2 kg is normally sufficient for radioactivity analysis. This sample may be collected in perforated polythene bags and stored under refrigeration. Sample weight should be taken at the earliest before storage. Vegetables and vegetation vary from place to place and the sample chosen need to be representative for the location.

The sample can be dried at 110°C in an oven, after removing non-edible portions. Dry sample should be powdered for the purpose of gamma spectrometric analysis. The dry powder can be ashed in a muffle furnace at 450 ± 10°C and can be taken for radiochemical analysis. Wet ashing with nitric acid (HNO_3) and hydrogen peroxide (H_2O_2) can also be carried out before radiochemical analysis. Sample weight, dry weight, and ash weight can be noted.

4.4.5 *Food Crops*

Rice, wheat, millets and pulses are the main food crops in the country. Samples may be collected from the field or from the granaries where grain is stored. About 2 kg of sample normally be sufficient. Dry crop sample can be directly counted without processing. For radiochemical analysis, the crops have to be either dry ashed in muffle furnace at 500°C or wet ashed using nitric acid (HNO_3) and hydrogen peroxide (H_2O_2).

4.4.6 *Milk*

Milk needs to be collected from dairy farms where milk from different area is processed for distribution. In the absence of a dairy, sample should be collected from about five milk producers and composited to make a representative sample from the area. Milk sample should be preferably taken from cattle which graze in the outside grass land and not from cattle fed from stored cattle feed. One litre of milk is normally be adequate for radioactivity measurement.

Refrigeration method is sufficient for short time preservation of milk samples. Long term preservation can be carried out by adding Formalin (about 5 ml of 5 percent solution per liter) as a preservative. Milk can be ashed below 450+10°C for the purpose of radiochemical analysis. A direct gamma spectrometry using NaI:Tl detector can also be carried out.

4.4.7 *Meat/Animal Thyroid*

Fresh meat of cattle, sheep or goats may be collected from the local abattoir or from the local market. Non-edible portion should be removed and only edible portion has to be processed and analyzed. Bones are easily separated from the flesh by heating the sample at 150°C in an oven for 30 min to 1 h. Meat samples can be collected in plastic bags and stored in deep freezer after taking weight. Normally 2 kg of soft tissue should be sufficient.

Animal thyroids have to be collected mainly from the place where animals are slaughtered for meat. The person collecting this sample needs to know the position of thyroid gland in animal body. Thyroids can be kept in polythene specimen tube and can be stored by refrigeration.

The meat samples may be ashed below 450°C to avoid loss of radio cesium at higher temperatures. Ashing can be done without ignition of the sample. 10 to 25 g of ash is sufficient for radiochemical analysis.

4.5 Samples from Aquatic Environment

4.5.1 *Fresh Water/Sea Water*

In case of municipal supplies, drinking water sample can be collected from water taps. About 10-20 l of water can be collected in a plastic carboy and about 10 ml of concentrated HCl/l of sample can be added as preservative.

If well water has to be collected, water sample from the public well used by maximum number of people, need to be collected. Sample should be acidified with HNO_3 for preservation. Sampling river or lake water can be carried out from a bridge or dock or jetty or a boat. A plastic bucket tied to a nylon rope can be used to take the sample from the surface. Sample can be filled in a 5 l plastic carboy. Acidify with concentrated HNO_3 (5 ml/l) for sample preservation. For depth sampling special depth samplers are available (Nansen bottle sampler).

For sampling sea water from different depths, special depth samplers should be used. Continuous water samplers (with varying sampling time and varying sampling frequency) are available which can be used conveniently for sampling streams receiving liquid waste. While sampling of river water, lake or coastal waters, sample need to be collected slightly away from the shore where depth of water is 0.5 m or more. 5 to 10 l sample are normally sufficient to detect any sea water contamination. Concentrated HNO_3 (3 ml/l) can be added as preservative.

4.5.2 *Sediment*

River bed, lake bed and sea bed can be sampled using a grab sampler. About 1 to 2 kg sediment need to be collected in plastic bags and can be stored under refrigeration.

When depth profile of radioactivity in a sediment bed has to be investigated, a sediment core can be collected. A tube penetrates the sediment layer to take vertical cylindrical samples. A non return valve arrangement stops the sample falling down while lifting up. After lifting, the sediment core can be pushed out of the tube and cut into different fractions for studying vertical pattern of radionuclide distribution.

Before carrying out analysis, the sediment sample should be passed through a 10 mesh screen to remove large stones and pebbles. If necessary, sample can be forced through the sieve with a rubber stopper. The sample can be dried in oven at 110°C, powdered, sieved through 70 mesh and analyzed by gamma spectrometry. The sample can be ashed at 450°C in a muffle furnace for radiochemical analysis.

4.5.3 *Fish and Other Aquatic Organisms*

Fish sample from the coastal area can be collected from the fish landing centres, or brought directly from the boats or trawlers. Samples of marine organisms such as crabs, oysters, clams and sponges are good concentrators of radionuclides and can many times serve as indicators for specific radionuclides, and hence need to be collected along with fish samples. Oyster samples are good for indicators for Zn-65 detection in the environment; sponge is the best indicator for Ni-63 and Co-60. These samples of aquatic organisms can be collected depending on their availability. About 1 kg of sample is normally sufficient. Plastic bags can be used for keeping the collected samples. Samples should be kept in an ice box soon after collection and subsequently stored in deep freeze, if it is not taken up for analysis immediately.

The sample may be processed to isolate edible and non edible portion and oven dried at 110°C. In case of aquatic organism such as fishes, clams, etc, heating at about 150°C in an oven helps in separating soft tissues from bone or shells. Dry ashing in a muffle furnace (at 450°C) or wet ashing with HNO_3 can be carried out for radiochemical analysis. Wet oxidation of soft tissue is better for preventing loss of volatile radionuclide.

4.5.4 *Aquatic Plants and Weeds*

The phytoplankton and zooplankton samples from the water body may be collected by passing a known volume of water through plankton net. The samples can be preserved at 4 percent formalin.

Sea weeds are good samples for I-131 detection in coastal waters and can be used as an indicator. Samples can be collected in plastic bags with perforations and preserved in deep freezer. Processing of these samples can be done similar to terrestrial vegetation. The important point to be observed in case of aquatic plant sample processing is to dislodge the deposited silt by thorough washing. About 0.5 to 1 kg sample is adequate for radionuclide measurement. Noting of sample weight, dry weight and ash weight is essential and need to be properly recorded.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Nuclear Materials for Peaceful Applications Sectional Committee, CHD 30

Organization	*Representives(s)*
Bhabha Atomic Research Centre (Radiochemistry & Isotope Group), Mumbai	Dr V. Venugopal (***Chairman***) Dr K. L. Ramakumar (*Alternate*)
Bhabha Atomic Research Centre (Environment Division), Mumbai	Dr (Shrimati) G. G. Pandit
Bharat Heavy Electricals Limited, Hyderabad	Shri K. Natarajan
Board of Radiation and Isotope Technology (BRIT), New Delhi	Dr N. C. Goomer
Defence Research & Development Organisation, Jodhpur	Shri S. G. Vaijapurkar Dr Pradeep Narayan (*Alternate*)
Department of Atomic Energy, Nuclear Fuel Complex, Hyderabad	Dr B. N. Murthy Shri Y. Balaji Rao (*Alternate*)
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Indian Rare Earths Ltd (Research Centre), Mumbai	Shri A. N. Patra Shri V. K. Verma (*Alternate*)
Indira Gandhi Centre for Atomic Research, Kalpakkam	Dr Baldev Raj Dr K. S. Viswanathan (*Alternate*)
Larsen and Tubro Ltd, Mumbai	Shri K. Kishan Babu Shri Anil Parab (*Alternate*)
Madras Atomic Power Station, Kalpakkam	Dr K. S. Krishna Rao
Ministry of Defence (Institute of Nuclear Medicine and Allied Sciences), Delhi	Dr Ravi Kashyap Dr Anupam Mondal (*Alternate*)
Ministry of Defence (DGQA), Pune	Shri C. S. P. Rathore Shri P. G. Date (*Alternate*)
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Tarapur Atomic Power Station (3 & 4), Mumbai	Shri S. K. Pawar Dr S. C. Dash (*Alternate*)
Uranium Corporation of India Ltd, Jharkhand	Shri S. Siddique
Vikram Sarabhai Space Research Centre, Thiruvananthapuram	Representative
In personal capacity, Lucknow	Shri S. P. Agarwal
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Member Secretary
Shrimati Meenal Passi
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